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» Energetic Materials

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Author: **Subject: Ammonium Perchlorate Synthesis**

TitusGabonicus
Harmless

posted on 3-8-2011 at 11:45

Ammonium Perchlorate Synthesis

Posts: 11
Registered: 11-5-2011
Location: UK
Member Is Offline

Mood: No Mood

I thought it interesting, the opinion danger level attached to the following reaction: {synthesis of AP, from 'Hawley's Chemical Dictionary'}
Reacting NH4ClO3 with NH4OH and HCL. It has little more detail, and for safety, I will expand no more.
I can see possible reaction mechanisms, producing AP + NH4Cl + NaCL + H2O.
Converting AP to HClO4, using HN03 + HCl, is an extensuion to the question.
If NG is 10 on the synthesis danger scale, how would you rate these reactions?

[Edited on 3-8-2011 by TitusGabonicus]

Scientists for the boycotting of Political assimilation

PROFILE FIND

hissingnoise
International Hazard

posted on 3-8-2011 at 11:55

Posts: 3922
Registered: 26-12-2002
Member Is Offline

Mood: Pulverulescent!

> **Quote:**
>
> Reacting NH4ClO3 with NH4OH and HCL. It has little more detail, and for safety, I will expand no more.

Just as well, since ammonium chlorate is dangerously sensitive!

PROFILE FIND

hissingnoise
International Hazard

posted on 3-8-2011 at 13:17

Posts: 3922
Registered: 26-12-2002
Member Is Offline

Mood: Pulverulescent!

AP is prepared by two methods - metathesis with NaClO₄ and neutralisation of HClO₄.

PROFILE FIND

AndersHoveland
Hazard to Other Members, due to

posted on 3-8-2011 at 14:39

repeated speculation and posting of untested highly dangerous procedures!

Posts: 1986
Registered: 2-3-2011
Member Is Offline

Mood: No Mood

One typically would want to reduce out any trace of chlorates, using 20%HCl, before they add an ammonium salt to precipitate out the NH4ClO4, so as to avoid formation of any unstable ammonium chlorate.

But you are asking what would happen if the ammonium chlorate was reacted with the HCl after. Do not really know.
Hydrogen peroxide actually preferentially attacks hydrochloric acid before it attacks ammonia. Simply mixing H2O2 and NH4OH will not result in any immediate reaction, whereas mixing H2O2 and HCl immediately results in formation of bubbles (vissible if 30% reactant concentrations are used). This demonstrates that nitrogen is a more "electronegetive" element than chlorine, despite the fact that formation of diatomic nitrogen is often very favorable. It would not be possible for NH3 to burn in air if not for the formation of the strong N-N triple bond. (for comparison, HCl gas cannot burn in air on its own).

The reduction of chloric acid by H2O2 has a very slow reaction rate unless heated, so it may be that, if reacting NH4ClO3 with HCl, the ammonium ions will generally be safe from direct oxidation. The only way to know for sure is to do the reaction, and see if any (and how much) nitrogen gas results. Of course, the chlorine generated will then form some of the dangerous NCl3 with the ammonium ions!

ClO3[-] + (6)HCl --> Cl[-] + (3)H2O + (3)Cl2

The reaction forming nitrogen trichloride is actually an equilibrium reaction:
NCl3 + (4)HCl <==> NH4Cl + (3)Cl2

The formation of NCl3 is only favorable within a certain pH range. Very low pH will prefer hydrolysis of NCl3. Alternatively, higher pH will prefer formation of chloramine instead.
If there is any free ammonia in the reaction, nitrogen gas will tend to form rather than NCl3.

So, yes, such a reaction would potentially be very dangerous, because some NCl3 would likely form in droplets at the bottom. If you do the reaction, use a plastic container (not glass which can shatter), wear protective eye goggles, and only experiment with very small quantities of reactants. Try to keep your fingers away from the bottom of the container, where the NCl3 could collect, and possibly explode without warning.

[Edited on 3-8-2011 by AndersHoveland]

I'm not saying let's go kill all the stupid people...I'm just saying lets remove all the warning labels and let the problem sort itself out.

PROFILE WWW FIND

Rosco Bodine
International Hazard

posted on 4-8-2011 at 08:43

Posts: 6335
Registered: 29-9-2004
Member Is Offline

Mood: analytical

Some serious fact checking should be done concerning just about every word in the above post by Anders Hoveland.

PROFILE FIND

AndersHoveland
Hazard to Other Members, due to repeated speculation and posting of untested highly dangerous procedures!

posted on 4-8-2011 at 10:39

Posts: 1986
Registered: 2-3-2011
Member Is Offline

All the above information has already been posted on this forum with references, but here is the information again:

Here is the information about the equilibrium of NCl3:
http://books.google.com/books?id=QQkSAAAAIAAJ&pg=PA2178&...

The reaction between solutions of chloric acid (HClO3) and hydrogen peroxide does not have any appreciable reaction rate until a temperatures above 70degC. (note that perchlorate is not a reaction product in the decomposition reaction, although it may be likely that traces are formed). experiments conducted by Sand, published in Zelt phys. Chem.,50, 465 (year 1904)

Mood: No Mood

A mixture of hydrogen peroxide and ammonium hydroxide (in a 1:3 ratio) acts as a reactive oxidizer, which can attack organic compounds and elemental carbon. The reaction rate is negligible at room temperature, but when heated to 60°C the reaction becomes vigorous and self-sustaining. Such solutions are sometimes known as "base piranha". With a 1:1:5 volume ratio of NH4OH, H2O2, and H2O, respectively, the half-life times of peroxide were 4 hours at 50°C and 40 minutes at 80°C. "Reaction of Ozone and H2O2 in NH4OH Solutions and Their Reaction with Silicon Wafers" Japanese Journal Applied Physics. 43 (2004) pp. 3335-3339.

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PROFILE WWW FIND

Rosco Bodine
International Hazard
posted on 4-8-2011 at 11:04

Posts: 6335
Registered: 29-9-2004
Member Is Offline

Mood: analytical

Using an elevated temperature will be the best precaution against nitrogen trichloride formation and against ammonium chlorate formation Your observations about electronegativity are incorrect. There is no appreciable reaction between H2O2 and HCl. Frankly it is difficult to even understand how H2O2 is relevant to the topic. Some fact checking would be in order here because you seem to be all over the place with your response that is not really an answer to the question being asked. The question being asked by the original poster relates to rating the danger of a reaction which does not take place ....therefore the original post is based upon a premise that is an oxymoron, to "rate the danger" of a reaction which does not occur.

Given that the reaction which is the premise for the question does not hold true,
then how can any of the answers not pointing that out be considered informed or intelligent answers?

[Edited on 4-8-2011 by Rosco Bodine]

PROFILE FIND

AndersHoveland
Hazard to Other Members, due to repeated speculation and posting of untested highly dangerous procedures!
posted on 4-8-2011 at 11:16

Posts: 1986
Registered: 2-3-2011
Member Is Offline

Mood: No Mood

> **Quote: Originally posted by Rosco Bodine**
>
> There is no appreciable reaction between H2O2 and HCl. Given that the reaction which is the premise for the question does not hold true,
> then how can any of the answers not pointing that out be considered informed or intelligent answers?

Not true.
http://www.sciencemadness.org/talk/viewthread.php?tid=14490

oxidation by H2O2 was used as a comparison to how acidified chlorate might react, whether it would prefer to oxidize chloride, or ammonium ions.

[Edited on 4-8-2011 by AndersHoveland]

I'm not saying let's go kill all the stupid people...I'm just saying lets remove all the warning labels and let the problem sort itself out.

PROFILE WWW FIND

Rosco Bodine
International Hazard
★★★★★

posted on 4-8-2011 at 11:25

Posts: 6335
Registered: 29-9-2004
Member Is Offline

Mood: analytical

Hilarious .....you give yourself as a reference. This is like answering the question "which came first, the chicken or the egg?" by saying in answer
"It isn't about which came first, but more about the number of fertile eggs appearing simultaneously at the moment of creation of laying hens, provided by the Lord as a chore for them to do of sitting on the nest when not pecking corn"

Obviously, God did not want chickens to suffer from boredom.

By the way, recompiling my words out of context is a really neat trick .....but it is deceptive in that it obscures the meaning of what I said. Hey Anders, most of us here reside on a planet called Earth. If not too much trouble, could you please join us here and knock off the B.S. with off the cuff misinformation and pseudoscientific jibberish which just keeps getting you caught at every turn? You are too often speaking authoritatively and incorrectly in what you are saying.

[Edited on 4-8-2011 by Rosco Bodine]

PROFILE FIND

AndersHoveland
Hazard to Other Members, due to repeated speculation and posting of untested highly dangerous procedures!
★★★★★

posted on 4-8-2011 at 12:35

Posts: 1986
Registered: 2-3-2011
Member Is Offline

Mood: No Mood

> **Quote:** Originally posted by Rosco Bodine
>
> Hilarious .....you give yourself as a reference. This is like answering the question "which came first, the chicken or the egg?"
>
> By the way, recompiling my words out of context is a really neat trick .....but it is deceptive in that it obscures the meaning of what I said. Hey Anders, could you knock off the B.S. with off the cuff misinformation and pseudoscientific jibberish which just keeps getting you caught at every turn?

What are you talking about? Yes, the link refered to one of my prior posts, but references *were* given in that post for the reaction of H2O2+HCl.
(Livingston and Bray, J. American Chem. Society, Volume 47, p2069 (1925) and "Oxidation of Hydrogen chloride with hydrogen peroxide in aqueous solution" V.I. Skudaev, A.B. Solomonov)

It can be seen on the Pauling electronegativity scale that nitrogen (value of 3.04) has almost the same value as chlorine (3.16). http://www.tutor-homework.com/Chemistry_Help/electronegativi...
Of course, these values to do not correlate to how the elements will behave in all reactions, or what their chemical reactivities at ordinary temperatures are, but it is at least somewhat indicative.

From two of the above mentioned reactions, it would seem that hydrogen peroxide more readily oxidizes hydrochloric acid than ammonia. But of course, the chlorine which forms gets reduced back as fast as it forms.

References have been provided for all the reactions that were mentioned. If there are any other reactions that you would like to have references for, just ask.

Certainly was not trying to be deceptive or misquote you. How do you even perceive that?

[Edited on 5-8-2011 by AndersHoveland]

I'm not saying let's go kill all the stupid people...I'm just saying lets remove all the warning labels and let the problem sort itself out.

PROFILE WWW FIND

Rosco Bodine

posted on 4-8-2011 at 13:13

International Hazard
★★★★★

Posts: 6335
Registered: 29-9-2004
Member Is Offline

Mood: analytical

You recompiled my words to misquote me as if H2O2 was the issue instead of an original post that was about a reaction which isn't a reaction.

You are still beating the dead horse H2O2 aspect which is irrelevant.
And I did not say there was no reaction.....I said there was no appreciable reaction, as in no significant reaction ratewise or otherwise readily apparent.
You are not speaking in terms of practical reaction schemes or rates as would be applicable to a realistic process, if the presumed reaction of the original post was valid and relevant ....which it is not.

Everything that follows and derives from that incorrect premise is revealing.

If you think you elevate yourself to post a hate blog against me have at it, if you want to descend to a new low. You have come here to a forum where intelligent people try to carry on intelligent discussions which are based in real science fact, and you take a condescending tone towards readers of this forum as if you were somehow the knowledgeable professor, but what you say is not of a quality which bears out the presumption you clearly have about the vastness of your own knowledge. Consequently you often state things that are just flat wrong and it is unscientific and certainly unprofessional. If you read a journal article with some comprehension, you then take giant leaps in estimating the scope of your own knowledge and it plainly shows. I don't want to argue with you. I would just ask that you stop posting a lot of things that you don't know but only think without any differentiation between the two. The decorum around here is not rigid with regards to discussion, but this is a science forum not an anything that can be imagined should be posted for fact kind of blog, where it then becomes the job of the rest to scratch their heads and wonder how much is truth or fiction.

PROFILE FIND

AndersHoveland
Hazard to Other Members, due to repeated speculation and posting of untested highly dangerous procedures!
★★★★★

posted on 4-8-2011 at 13:56

Question for TitusGabonicus,
just to clarify, did the entry in 'Hawley's Chemical Dictionary' specifically say that reacting NH4ClO3 with NH4OH and HCL would make ammonium perchlorate?

It does not seem like any perchlorate would be formed, but cannot be completely sure.

Posts: 1986
Registered: 2-3-2011
Member Is Offline

Mood: No Mood

[Edited on 5-8-2011 by AndersHoveland]

I'm not saying let's go kill all the stupid people...I'm just saying lets remove all the warning labels and let the problem sort itself out.

PROFILE WWW FIND

Rosco Bodine
International Hazard
★★★★★

posted on 4-8-2011 at 14:31

Exactly.

Posts: 6335
Registered: 29-9-2004
Member Is Offline

Mood: analytical

PROFILE FIND

TitusGabonicus
Harmless
★

posted on 16-8-2011 at 11:41

The 'Hawley edition' of 35 years ago, reads as does its11th..edition.(1984)
Derivation: (Re. NH4ClO4): Interaction of ammonium hydroxide, hydrochloric acid and sodium

Posts: 11

Registered: 11-5-2011
Location: UK
Member Is Offline

Mood: No Mood

chlorate. Recovery by crystallizatuion.
Sounds scary, its true. I therefore am questioning the accuracy of this procedure. It may be an error? Thus, I placed it to you, to evaluate its validity. Thank you.

PROFILE FIND

quicksilver
International Hazard
★★★★★

posted on 16-8-2011 at 12:58

Posts: 1820
Registered: 7-9-2005
Location: Inches from the keyboard....
Member Is Offline

Mood: ~-=SWINGS=-~

> **Quote: Originally posted by TitusGabonicus**
>
> The 'Hawley edition' of 35 years ago, reads as does its11th..edition.(1984) Derivation: (Re. NH4ClO4): Interaction of ammonium hydroxide, hydrochloric acid and sodium chlorate. Recovery by crystallizatuion. Sounds scary, its true. I therefore am questioning the accuracy of this procedure. It may be an error? Thus, I placed it to you, to evaluate its validity. Thank you.

Welcome. Nice first post. I'm glad you used documented source & it is a question many would have, were they to read the synthesis as presented.
Would you happen to have a scan or copy of the exact synthesis? {Are you sure of the typed example?} Did the material mention temperature(s) control of the process? And / or control of reactions w/ hydroxide?

I think we're all on the same page in so far as the dangerous nature of ammonium chlorate (in situ or not) during this synthesis but did the author delve into methods to control the reaction, or caveats regarding manipulation so as to place a level of control therein? Was **any** further background given? Occasionally broadly expressed methods are used as means of learning extraneous issues & not as synthesis practicum.

NOTE: Often material available in the UK is tough to find in the USA, especially if you're looking at classroom text.

PROFILE FIND

dann2
International Hazard
★★★★★

posted on 16-8-2011 at 14:06

Posts: 1523
Registered: 31-1-2007
Member Is Offline

Mood: No Mood

It's simply an error in the book. Jade Ledgard mush have been reading it too.....

http://oxidizing.110mb.com/chlorate/further/cep1957/cep1957....
http://oxidizing.110mb.com/chlorate/further/cep1961/cep1961....

Ammomium Hydroxide + HCl gives Ammonium Chloride + water.
Ammonium Chloride + Sodium Perchlorate (not Chlorate!) gives NaCl + Ammonium Perchlorate (all in water solution)
Crops of Ammonium Perk, must be crystallized out of the solution. Crops of NaCl will also be taken out at the proper times (continous production).

Dann2

[Edited on 16-8-2011 by dann2]

PROFILE FIND

AndersHoveland
Hazard to Other Members, due to repeated speculation and posting of untested highly dangerous procedures!
★★★★★

posted on 16-8-2011 at 16:40

> **Quote: Originally posted by dann2**
>
> It's simply an error in the book.

Posts: 1986
Registered: 2-3-2011
Member Is Offline

Mood: No Mood

Yes, it is to be strongly suspected that the book made a mistake. Ammonium *chlorate* is more likely to result from this procedure, assuming excess HCl is not used.

I'm not saying let's go kill all the stupid people...I'm just saying lets remove all the warning labels and let the problem sort itself out.

PROFILE WWW FIND

TitusGabonicus
Harmless

posted on 17-8-2011 at 02:17

Posts: 11
Registered: 11-5-2011
Location: UK
Member Is Offline

Mood: No Mood

I too, think it is an error.
Hawley has been one of my 'desert island books', for many years.
It is a marvellous text, but you could still lose fingers, lest you triplecheck reactions from ANY source. Least of all when dealing with (per)chlorates.
My thanks to those who contributed.
If 'Hawley' has any comment, I'm sure it would be well received.

PROFILE FIND

Hennig Brand
International Hazard

posted on 21-8-2011 at 09:35

Posts: 1284
Registered: 7-6-2009
Member Is Offline

Mood: No Mood

This is from the same website that dann2 linked to, very good website. It has a method for making ammonium perchlorate starting with the commonly available potassium perchlorate. It seems like it would work, and not be all that difficult either. Ammonium sulfate is a very common/cheap fertilizer which is easily obtained.

I made 500g of potassium perchlorate with the help of that website over a year ago. I like that website.

Attachment: Ammonium Perchlorate from Potassium Perchlorate.docx (15kB)
This file has been downloaded 696 times

Edit:
After reading the thread a bit more carefully I realize I am not really on topic. I will leave it anyway, as I think it is interesting.

[Edited on 21-8-2011 by Hennig Brand]

PROFILE FIND

dann2
International Hazard

posted on 21-8-2011 at 13:51

Posts: 1523
Registered: 31-1-2007
Member Is Offline

Mood: No Mood

@HB
Stop, stop you making me blush FFS!!!!!!!!!

PROFILE FIND

Hennig Brand
International Hazard

posted on 22-8-2011 at 05:31

Posts: 1284
Registered: 7-6-2009
Member Is Offline

Sorry about that. You blush easily do you?

So that is your website then. I was never 100% sure, but I did get the impression that you were

Mood: No Mood

somehow connected to it. Well, if it makes you uncomfortable I'll lay off the compliments, but it still is a nice collection of information on the topic.

PROFILE FIND

WillCourney
Harmless

posted on 30-12-2017 at 06:19

Posts: 1
Registered: 30-12-2017
Member Is Offline

Mood: No Mood

It would be a good topic for my next coursework. I'll learn more about it and will start to write with some service's help.

Do My Math Homework @ Professional Experts Online Help

PROFILE FIND

Laboratory of Liptakov
International Hazard

posted on 1-1-2018 at 04:08

NH4ClO4

Posts: 818
Registered: 2-9-2014
Member Is Offline

Mood: cool.gif

Ammonium Perchlorate from Potassium Perchlorate.docx (15kB) One from best information about NH4ClO4 preparation ever. Genious method.

Safety explosive Alfred Nobel 1867. Safety ecologic detonator Dr. Liptakov 2015.

PROFILE FIND

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» Energetic Materials